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DISLOCATION DYNAMICS IN VANADIUM: A NUCLEAR MAGNETIC RESONANCE AND TRANSMISSION ELECTRON MICROSCOPIC STUDY

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Abstract—Pulsed nuclear magnetic resonance proved to be a complementary new technique for the study of moving dislocations in b.c.c. metals. From the motion induced part of the spin-lattice relaxation rate the mean jump distance of mobile dislocations has been measured in Vanadium as a function of temperature. The NMR experiments are combined with transmission electron microscopic investigations to reveal the static structure of defects in the samples. The NMR experiments show that the mean jump distance is nearly constant below 230 K whereas it decreases substantially above 230 to 300 K indicating a transition that marks two different mechanisms. NMR observations in combination with TEM support the physical picture that above the transition temperature dislocation segments are stopped between localized obstacles whereas below T_c the lattice friction controls the plastic behaviour.

Résumé—La résonance magnétique nucléaire pulsée s'est révèlée une nouvelle technique complémentaire pour étudier le mouvement des dislocations dans les métaux CC. A partir de la partie induite par le mouvement de la vitesse de relaxation du spin du réseau, la longueur du saut moyen des dislocations mobiles est mesurée dans le vanadium en fonction de la température. Les expériences de RMN sont combinées avec des observations par microscopie électronique en transmission pour révèler la structure statique des défauts dans les échantillons. Les expériences de RMN montrent que la longueur du saut moyen est presque constante au dessous de 230 K tandis qu'elle décroit substantiellement au dessus de 230 K jusqu'à 300 k indiquant une transition qui marque deux mécanismes différents. Les observations de RMN en liaison avec la MET s'accordent à montrer qu'au dessus de cette température de transition les segments de dislocations sont arrêtés entre les obstacles localisés tandis qu'au dessous de T_c le frottement du réseau contrôle le comportement plastique.

Zusammenfassung—Gepulste Kernspinresonanz hat sich als eine komplementäre neue Methode zur Untersuchung sich in krz. Metallen bewegender Versetzungen bewährt. Aus dem von der Bewegung induzierten Teil der Spin-Gitter-Relaxationsrate wird die mittlere Sprungweite beweglicher Versetzungen in Vanadium in Abhängigkeit von der Temperatur gemessen. Die Experimente werden kombiniert mit durchstrahlungselektronenmikroskopischen Beobachtungen, um die statische Defektstruktur aufzuklären. Den Kernspinexperimenten zufolge ist die mittlere Sprungweite unterhalb 230 K näherungsweise konstant, wohingegen sie oberhalb 230 K bis 300 K beträchtlich absinkt, welches auf einen Übergang zu einem anderen Mechanismus hinweist. Die Beobachtungen im Durchstrahlungselektronenmikroskop belegen das physikalische Bild, daß oberhalb dieser Übergangstemperatur Versetzungssegmente an lokalisierten Hindernissen aufgehalten werden. Unterhalb T_c steuert die Gitterreibung das plastische Verhalten.

1. INTRODUCTION

Plastic deformation of b.c.c. metals in general exhibits characteristic features at low temperatures [1], e.g. a strong temperature dependence of the yield stress and anomalous slip. The latter has been studied most extensively in α-Fe and in Nb, however, this slip mode has been observed in other b.c.c. metals as well, such as Ta [2–5], V[6–8] and Mo [9, 10]. The occurrence of the slip is anomalous as the slip plane observed belongs to one of the {110} planes with a small Schmid factor [11–16].

In the past, *in situ* TEM experiments have been performed [17, 18] at low and intermediate temperatures on Nb, Mo and α-Fe. In all these b.c.c. materials a transition has been observed in the dislocation dynamics between a low temperature behaviour, characterized by an almost continuous movement of long screw dislocations and an intermediate temperature behaviour, where mixed dislocations predominate. The present paper reports on the dislocations dynamics measured using a complementary new technique for b.c.c. metals, i.e. pulsed nuclear magnetic resonance as a function of temperature. The great strength of nuclear magnetic resonance is that the resonance signal is characteristic of the particular nucleus studied. Moreover, the surrounding of a nucleus may affect NMR properties like relaxation time. Accordingly, NMR can be used to study the environment of the nuclei providing microscopic information of atomic motion. In contrast to *in situ* transmission electron microscopic

observations of dislocation motion, NMR is detecting more of the bulk of the material and does not allow only investigation of dislocations near free surfaces, like in TEM where the existence of image forces may cause their behaviour very different from that in the interior of the material.

2. EXPERIMENTS

In this investigation we focus on plastic deformation experiments at a constant strain rate $\dot{\epsilon}$. This type of situation is governed by Orowan's equation [19], where the strain rate is linearly proportional to L/τ_m. Here L is the mean jump distance of dislocations and τ_m represents the mean time of stay of a dislocation in front of an obstacle. In the past we showed that pulsed nuclear magnetic resonance (NMR) is a useful tool for studying dislocation motion under constant strain rate conditions. It turned out that the mean jump distance of dislocations could be deduced from these measurements. For detailed information of this technique, reference is made to our review paper [20]. Only a concise summary will be presented here.

The method is essentially based on the interaction between nuclear electric quadrupole moments and elastic-field gradients at the nucleus. Around a dislocation in a cubic crystal the symmetry is destroyed and interactions between nuclear electric quadrupole moments and electric-field gradients arise. Whenever a dislocation changes its position in the crystal, the surrounding atoms have also to move, thus causing time fluctuations of the quadrupolar spin Hamiltonian for spins with $I > \frac{1}{2}$. Furthermore, for the investigation of rather infrequent defect motions as in the case of moving dislocations, the spin-lattice relaxation time in a weak rotating field, $T_{1\rho}$, has proved to be the most appropriate NMR parameter affected by such motions because the "time windows" of $T_{1\rho}$ lie in the range of τ_m.

Using sufficiently deformation rates $\dot{\epsilon}$, so that $1/\tau_m \ll \omega$, the Larmor frequency in the rotating frame, the dislocation-induced relaxation rate is given by

$$\frac{1}{T_{1\rho}} = \frac{A_Q}{H_1^2 + H_{L\rho}^2} \frac{1}{bL} \dot{\epsilon} \qquad (1)$$

where A_Q depends on the mean-squared electric-field gradient due to the stress field of a dislocation of unit length and the quadrupolar coupling constant. Here $H_{L\rho}$ is the mean local field in the rotating frame and H_1 is the (weak rotating) applied field; b represents the magnitude of the Burgers vector. Since A_Q and $H_{L\rho}$ can be determined separately, for a given plastic deformation rate $\dot{\epsilon}$ the spin-lattice relaxation rate can be used to determine L as a function of a physical parameter as strain. It has to be emphasized that the observed spin-lattice relaxation rate is a sum of $1/T_{1\rho}$ [equation (1)] and a background relaxation rate $1/T_{1\rho}^0$. The background relaxation that is caused in metallic samples by conduction electrons (Korringa relaxation) can easily be determined be setting $\dot{\epsilon} = 0$ in the actual NMR experiment.

The applied strain rate $\dot{\epsilon}$ is high. This is necessary because of the following reason:

The spin-lattice relaxation rate $T_{1\rho}$ is, in principle, a function of the quadrupolar "lattice correlation function" and the "quadrupolar spin-correlation function" $K_Q(t)$. The former depends explicitly on the number of mobile dislocations and the electric field gradient at an atomic site. The latter describes a thermal mixing process between the nuclear spins following a dislocation jump. This process is determined by the overlap of the nuclear energy levels and characterized by a thermal mixing time T_m. When the mean time between successive movement of dislocations $\tau_m \gg T_m$, the lattice correlation function remains unchanged while $K_Q(t)$ decays to zero during quadrupolar thermal mixing. In practice, $K_Q(t)$ vanishes at all times when $\dot{\epsilon} \cong 1.s^{-1}$ and one obtains equation (1). In other cases the lattice correlation function may decay more rapidly than $K_Q(t)$ by which equation (1) becomes too complicated. Strictly speaking the assignment of a single spin temperature to the entire spin Hamiltonian then breaks down. Consequentially, these experiments are only meaningful in the analysis presented here when carried out at high strain rates of about $1\,s^{-1}$.

Polycrystalline samples of V(3N) with a grain size of the order of 100 μm were used. To avoid skin effect distortion of the NMR signal, the actual NMR experiment was carried out on a single rectangular foil of size $27 \times 12\,mm \times 100\,\mu m$. The starting material was cut by spark erosion to the sample size mentioned before and annealed at 1000°C for 1 h.

The NMR experiments discussed here are carried out between 100 and 330 K and the samples under investigation are plastically deformed at a constant strain rate in the range of $0.4–3.6\,s^{-1}$ by a servo-hydraulic tensile machine [21]. While the specimen is deforming the nuclear spin relaxation rate $T_{1\rho}^{-1}$ of ^{51}V is measured at an operating frequency of 15.7 MHz using the spin-locking technique [20]. Simultaneously the actual deformation ϵ is observed. Before and after each experimental run, the background relaxation rate is measured by setting $\dot{\epsilon} = 0$.

Transmission electron micrographs are taken by using a JEM 200 CX. Disk-type specimens are taken from the deformed foils by spark cutting to minimize deformation. The samples are electrochemically thinned in a polishing equipment at room temperature in a solution of 15% water and 85% perchloric acid.

3. RESULTS

In Fig. 1 the dislocation-induced contribution to the relaxation rate [equation (1)] measured with a constant locking field H_1 of 10 G is plotted as a function of plastic strain rate $\dot{\epsilon}$ (at $T = 295$ K). The slope of the curve up to $\dot{\epsilon}$ of about $10\,s^{-1}$ is found

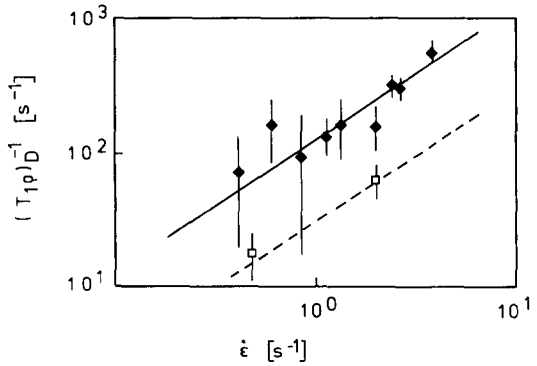

Fig. 1. Dislocation induced nuclear spin-relaxation rate vs strain rate $\dot{\epsilon}$ of ^{51}V in vanadium (solid line) and of ^{27}Al in aluminium (dahsed line).

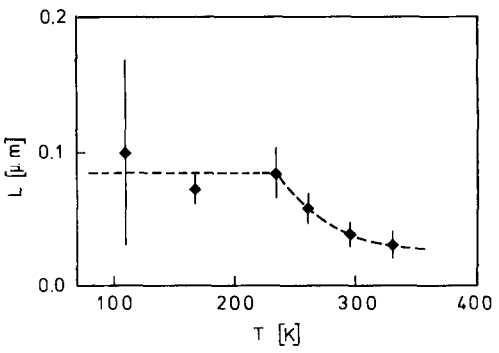

Fig. 2. Mean jump distance L of dislocations in vanadium (3 N) as a function of deformation temperature (at $\epsilon = 15\%$).

to be proportional to the strain rate as predicted by equation (1). From the magnitude of the ratio $(1/T_{1\rho})_D/\dot{\epsilon}$ the mean jump distance L can be determined if the other parameters in equation (1) are known.

The local fields $H_{L\rho}$ can be determined by measuring $(T_{1\rho})$ vs the locking field H_1. According to equation (1) a plot of the dislocation induced contribution to $T_{1\rho}$ vs the square of the locking field will yields a straight line which can be extrapolated to find the abscissa-intercept at $H_1^2 = -H_{L\rho}^2$.

In addition $H_{L\rho}$ can be experimentally determined by measuring the ^{51}V spin echo signal and fitting this to the second moments of the dipolar en quadrupolar broading functions, Δ_D^2 and Δ_Q^2, respectively and taking $3H_{L\rho} = \Delta_D^2 + \Delta_Q^2$. The various contributions to the local fields are listed in Table 1 at various temperatures.

As discussed in previous papers [22, 23], the coupling strength A_Q [equation (1)] of a dislocation of unit length can be determined in principle either theoretically or derived from experiments. However, since the value of the gradient-elastic constant which is the most crucial parameter affecting A_Q, is not known, the coupling strength A_Q has been estimated by: $A_Q = H_{Q\rho}^2/\rho_t$ [24]. Assuming a total dislocation density ρ_t of 10^{10} cm^{-2} after a deformation of 15% at 166 K and using $H_{Q\rho}^2 = (20 \pm 5)$ G^2 (Table 1) one yields $A_Q = (10 \pm 5)$ G^2.

From the magnitude of the slope of the curve $(T_{1\rho}^{-1})_D$ vs $\dot{\epsilon}$ the mean jump distances L have been determined between 100 and 330 K. The results are depicted in Fig. 2. Special attention has been paid to the typical inhomogeneity of the plastic deformation of b.c.c. materials (Lüders band formation). This has been worked out in more detail in Appendix I. The strain dependence of L has been obtained as well from measuring $T_{1\rho}^{-1}$ as a function of strain ϵ. Each data point depicted in Figs 1 and 2 represent the averaged value of five measurements.

Table 1. Local fields of ^{51}V in vanadium after 15% deformation at different temperatures T

T(K)	$H_{L\rho}^2$(G^2)	$H_{L\rho}$(G)	$H_{D\rho}$(G)	$H_{Q\rho}$(G)
166	28.3 ± 5	5.3 ± 0.5	2.8 ± 0.1	4.5 ± 0.6
233	22.2 ± 5	4.7 ± 0.5	2.8 ± 0.1	3.8 ± 0.6
260	19.5 ± 5	4.4 ± 0.6	2.8 ± 0.1	3.4 ± 0.7
295	18.1 ± 5	4.2 ± 0.6	2.8 ± 0.1	3.2 ± 0.8
330	16.3 ± 5	4.0 ± 0.6	2.8 ± 0.1	2.9 ± 0.9
Undeformed	10.2 ± 5	3.2 ± 0.8	2.8 ± 0.1	*ca* 1.5

The flow stress of the vanadium foils as a function of temperature is displayed in Fig. 3. Apparently the mean jump distance L measured by NMR is nearly constant below 230 K whereas it decreases considerably above 230 K. From Fig. 3 it is seen that the critical temperature of the 3N-Vanadium samples is about 300 K, i.e. higher than the onset of the decrease of the mean jump distance (Fig. 2).

In Fig. 4 typical transmission electron micrographs are presented of V deformed at 166 and 295 K. After plastic deformation at low temperatures [Fig. 4(a)] long primary screw dislocation parallel to [111] and dislocation tangles and loops are observed. After deformation at higher temperature [Fig. 4(b)] dislocations are accumulated in multipole bundles perpendicular to the glide direction. These bundles or dislocation walls run parallel to the [1$\bar{2}$1] line direction of primary edge dislocations and have a thickness of about 0.5 μm. Screw dislocations are present between those dislocation walls. The corresponding histograms of the interspacing distances between the dislocations are illustrated in Fig. 5(a,b), respectively. Both show a Poisson-like distribution with most

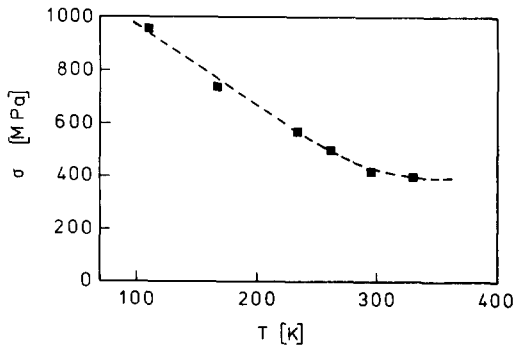

Fig. 3. Flow stress as a function of deformation temperature ($\dot{\epsilon} = 3.6 s^{-1}$, $\epsilon = 3\%$).

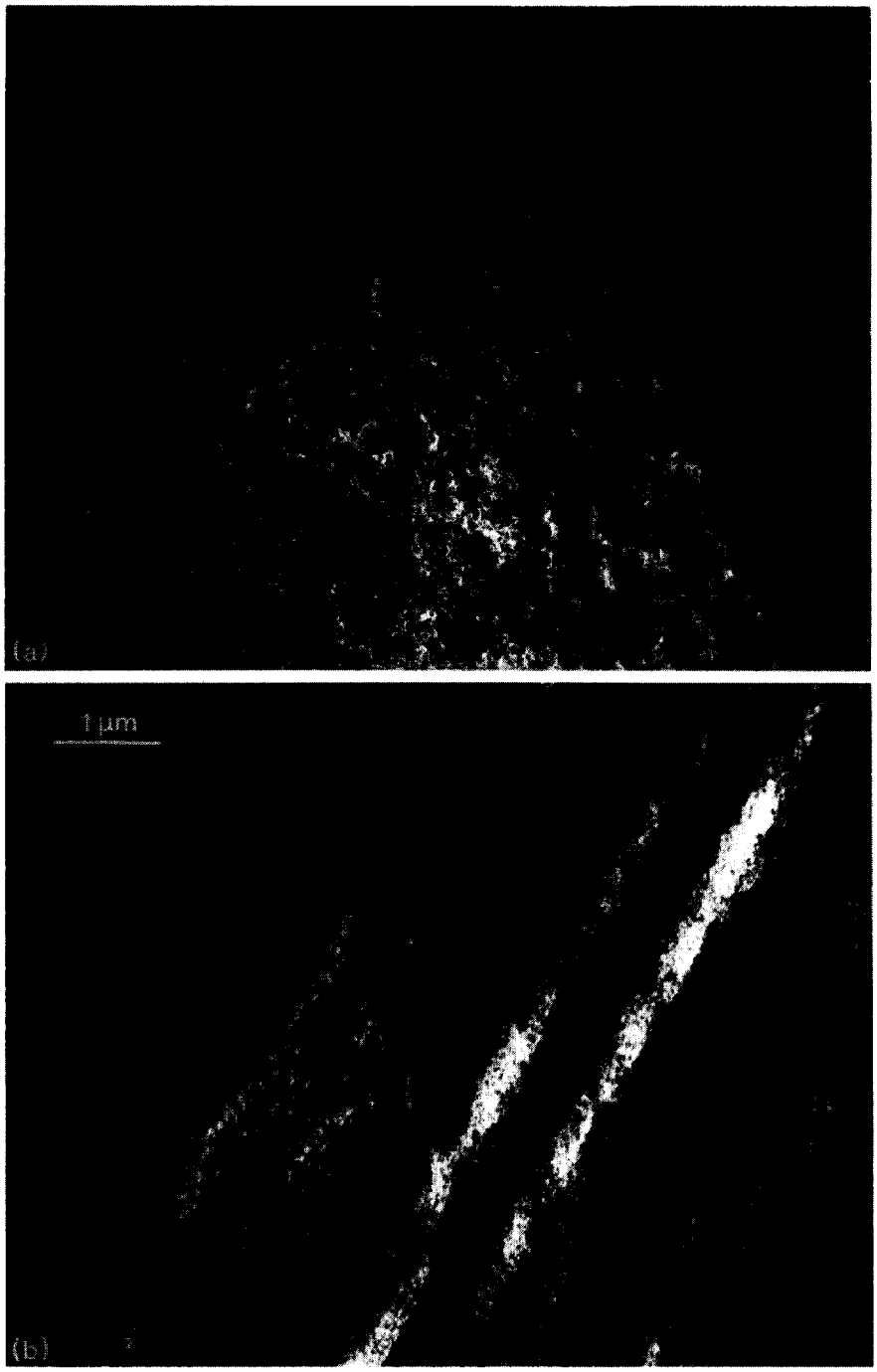

Fig. 4. Transmission electron micrograph of V deformed at $T = 166$ K (a) and $T = 295$ K (b). $\mathbf{g} = [200]$.

probable values of 0.07 and 0.05 μm for the interspacing distances between dislocations in V deformed at 166 K and at 295 K, respectively. The values obtained from the histograms based on TEM observations agree fairly well with the mean jump distance obtained from NMR experiments (Fig. 2). Hence, the dislocations produced by the plastic deformation process act as barriers for the movement of mobile dislocations. When dislocations are delayed at each of the intersections with the forest dislocations during a period of time larger than 10^{-4} s, spin-lattice relaxation takes place. As a result $T_{1\rho}^{-1}$ is determined by waiting time at each intersection and consequentially by the distance between the dislocations.

4. DISCUSSION

The application of the NMR technique to moving dislocations is based on the relationship between the spin-lattice relaxation rate and Orowan's equation,

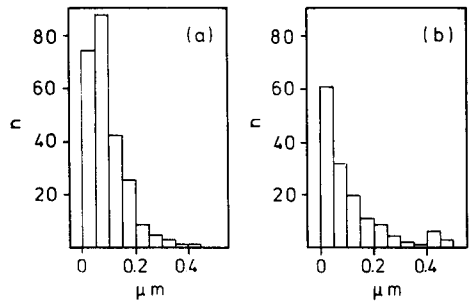

Fig. 5. Histograms of the distribution of interspacing distances between dislocations in V deformed at $T = 166$ K (a) and at $T = 295$ K (b).

where the strain rate is assumed to be linearly proportional to L/τ_m. The question rises how L should be interpreted. First, one should realize that the plastic strain rate $\dot{\epsilon}$ results from the movement of mobile screw dislocation density ρ_s with velocity V_s and a mobile edge dislocation density ρ_E with velocity V_E

$$\dot{\epsilon} = \rho_s b |\mathbf{V}_s| + \rho_E b |\mathbf{V}_s|. \quad (2)$$

In a real crystal there may exist contributions to $\dot{\epsilon}$ from dislocation loops whose edge or screw parts have been blocked. Consequently, ρ_s may depend on the mean free path in the crystal of edge dislocations, L_E, and the edge dislocation density on the screw mean free path, L_s. There is ample evidence from microscopic observations of b.c.c. metals at low temperature [25, 26] that the total screw dislocation density is much larger than the total edge dislocation density. Provided that this applies also to the mobile dislocation densities, and provided that $|\mathbf{V}_E|$ is not too much larger than $|\mathbf{V}_s|$ the term with $|\mathbf{V}_s|$ in (2) will predominate and consequently L in equation (1) should be interpreted as the mean slip distance of screw dislocations.

At low temperature and under stress, most dislocations are observed to be of screw character. However, their mobility is much lower than that of mixed dislocations at any stress level [18] whereas at intermediate temperature ($\cong 100$ K) screw dislocations appears to move by a double kink mechanism, since the dislocations remain straight and screw whilst moving. On increasing temperature the screw dislocation character disappears leading to a temperature behaviour fairly similar to that of f.c.c. metals.

Although the NMR relaxation rate in polycrystalline material cannot be analyzed in separate contributions of edge- and screw dislocation segments (see [27] for single crystals) the measurements at low temperature suggest that the L value determined is rather the mean free path of edge or mixed segments or even that of kinks. If L_0 is the average distance between obstacles along a screw dislocation at low temperature and unpinning occurs when the screw dislocation has passed a distance x_c beyond this obstacle than: $L_0 x_c = \rho_F^{-1}$ assuming that these obstacles are forest dislocations with density ρ_F. The velocity of the screw dislocations then depends on the average distance L_0 between the localized obstacles, i.e. the mean distance between the double kinks [18].

x_c, the average separation between pinning points along the dislocation line is temperature dependent [28] (similar to the temperature dependence of the breaking angle of unpinning). This critical distance is observed to increase with temperature, i.e. when the thermal stress associated with the lattice friction decreases.

A transition temperature can then be associated with the increase of flexibility of dislocations. Below T_c screw dislocations move almost continuously and the lattice friction on screws controls the plastic behaviour. Above T_c, L_0 reduces to zero before unpinning and therefore the dislocation segments stop between localized obstacles. At the transition temperature the critical distance x_c associated with forest cutting is taken to be equal to half the critical distance x_s for dislocation source operation, related to the radius of curvature by

$$x_c = \frac{\alpha \mu b}{\tau(T_c)} \quad (3)$$

where α is a constant (≈ 0.7), μ the shear modulus and τ the shear stress at T_c. Here it is assumed that only forest hardening exists and frictional forces disappear ($T \cong 300$ K in Figs 2 and 3). It should be emphasized that V(3N) is not a very pure material. Interstitial impurities may affect the motion of screw dislocations even more than forest dislocations, especially at small strains. Using $\tau_c = 210$ MPa from Fig. 3 x_c is predicted to be 0.041 μm which is in good agreement with the mean jump distance determined by NMR, namely 0.038 μm.

5. CONCLUSIONS

Nuclear spin relaxation measurements in the rotating frame are shown to be a complementary tool for studying dislocation dynamics in b.c.c. metals. In particular the experiments provide information of the mean jump distance of mobile dislocations. The mean jump distance turned out to be nearly constant for deformation taking place below 230 K whereas it decreases considerably above 230 K. The NMR observations in combination with transmission electron microscopy support the physical picture that above this transition temperature dislocation segments are stopped between localized obstacles whereas below T_c the lattice friction controls the plasticity.

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APPENDIX

In principle equation (1) is valid only when homogeneous deformation takes place in the specimen. As a matter of course, at inhomogeneous deformation (Lüders band formation) the spin-lattice relaxation rate is not equal for all parts in the sample. Suppose over a length Δx_i the specimen deforms in tension with a certain strain rate $\dot{\epsilon}(i)$. The spin-lattice relaxation rate over Δx_i can be written as

$$\left.\frac{1}{T_{1\rho}}\right|^{i} = c \cdot \dot{\epsilon}(i) \tag{A1}$$

where c is a constant depending on L [equation (1)]. To measure $T_{1\rho}$, the nuclear magnetization is allowed to decrease in the presence of a locking field H_{loc} for some time τ, then H_{loc} is turned off and the initial height of the nuclear free induction decay signal $S(t)$ is measured

$$S(\tau) = S(0)e^{-\tau/T_{1\rho}}. \tag{A2}$$

The height of S without deformation depends the fluctuation in the conduction electron-nucleus interaction leading to a relaxation rate $(1/T_{1\rho})^{\text{e}}$. During deformation a dislocation induced part contributes to the relaxation rate $(1/T_{1\rho})_{\text{D}}$. Therefore the nuclear free induction decay signal can be written as [20]

$$S' = S_0 \Sigma_i x_i \exp\left[-\tau\left\{\left.\frac{1}{T_{1\rho}}\right|^{\text{e}} + \left.\frac{1}{T_{1\rho}}\right|^{\text{D}}\right\}\right]. \tag{A3}$$

Combination of equation (A3) with equation (A2) and equation (A1) yields for $\Delta x \to 0$ and $\Sigma_i \Delta x_i = 1$

$$\frac{S'}{S} = \int_0^1 \exp[-\tau \cdot c \cdot \dot{\epsilon}(x)]\,\mathrm{d}x. \tag{A4}$$

According to equation (A4) the constant c (that contains the mean jump distance) can be calculated numerically for an inhomogeneous deformation. To a first approximation, assuming that the sample is deforming homogeneously within a fraction f, equation (A4) can be simplified by

$$\left.\frac{1}{T_{1\rho}}\right|^{\text{D}} = \frac{1}{\tau}\ln\left(\frac{f \cdot S}{S' + (f-1)S}\right). \tag{A5}$$

Based on optical observation it turned out that $f \approx 0.4$ for $\epsilon = 3\%$ deformation at 77 K.